

TestAmerica St. Louis SOP No. ST-RC-0039, Rev. 6 Effective Date: 07/31/2014

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Title: RADIUM-226 BY ALPHA SPECTROMETRY

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1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of radium-226 by direct measurement of alpha emissions using alpha spectrometry. It is applicable to liquid or other media where complete dissolution and carrier exchange are readily achievable in the laboratory.

1.2 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Radium isotopes are collected by ceprecipitation of radium with barium and lead sulfate and purified by precipitation from DTPA solution. The precipitate is washed with concentrated nitric acid followed by a water wash. The barium and lead sulfate precipitate is chelated using DTPA and the lead is removed using ammonium sulfide precipitation. Barium and radium acc-precipitated as a sulfate and collected on a 0.1 micron filter Radiometric yield is determined by gamma spectrometry using Ba-133 as a yield monitor and Ra226 activity concentrationis determined by alpha spectrometry.

3.0 **DEFINITIONS**

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Matrices which contain significant amounts of barium or strontium will have the effect of broadening the ra-226 peak (smeared spectrum) requiring expansion of the ROI.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
 - 5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Hydroxide Corrosive Corrosive Flammable Acetic Acid Corrosive Flammable Corrosive Flammable Corrosive Flammable Corrosive Flammable Flammable Corrosive Flammable Flammable Flammable Corrosive Acid Floson Corrosive Acid Acid Corrosive Acid Corrosive Acid Acid Corrosive Acid Acid Corrosive Acid Acid Corrosive Acid Acid Acid Corrosive Acid Acid	Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure	
Flammable Flambable Flamba		l l		chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye	
Acid Poison (Ceiling) inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Hydrofluoric Acid Corrosive Dehydrator Dehydrator Poison (TWA) Poison (STEL) Dehydrator Poison (STEL) Dehydrator Poison Carcinogen (TWA) Dehydrator Poison (STEL) Inhalation produces damage in inflammation of the nose, throat, and respiratory tract. Corrosive to the respiratory tract. Corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE. Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Sulfuric Acid Corrosive Oxidizer (TWA) Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurned vision, redness, pain and severe tissue burns. Can cause blindness.	Acetic Acid			Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of	
Acid Corrosive Dehydrator Dehydra	-			inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.	
Nitric Acid Corrosive Oxidizer Poison A ppm (STEL) Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Sulfuric Acid Corrosive Oxidizer Oxidizer Dehydrator Poison Carcinogen Carcinogen Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.	-	Corrosive		the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND	
Sulfuric Acid Corrosive Oxidizer (TWA) Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.	Nitric Acid	Oxidizer	(TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.	
1 – Always add acid to water to prevent violent reactions.		Oxidizer Dehydrator Poison Carcinogen	(TWA)	membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.	
2 – Exposure limit refers to the OSHA regulatory exposure limit. TWA – Time Weighted Average				tory exposure limit.	

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STEL – Short Term Exposure Limit

Ceiling – At no time should this limit be exceeded.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Analytical balance
- 6.2 Glassware as appropriate
- 6.3 Hot Plate
- 6.4 Mod Block
- 6.5 Centrifuge tubes, 50mL, conical
- 6.6 Centrifuge
- 6.7 Pipettes, various volumes
- 6.8 Syringe filters, Pall Acrodisk, 0.45u, or equivalent
- 6.9 Syringe, 20 nL, with Luer-Lok tip
- 6.10 Filters, Eichrom Resolve, polypropylene, 25mm dia., 1 μm; or equivalent
- 6.11 Suction filtering apparatus
- 6.12 Petri dishes, appropriate size to contain final prepared filters
- 6.13 Disks, flat plastic with double sided tape. F2 disks, plastic, withdouble sided tape from A.F Murphy Co.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Distilled or deionized water, ASTM II (1991) from the Milbore unit.
- 7.3 Acetic acid, 17.4N: glacial CH₃COOH (concentrated), specific gravity 1.05, 99.8%.
- 7.4 Acetic acid, 50%: to 50 mL of DI water add 50 mL of glacial acetic acid.
- 7.5 Ammonium hydroxide 15N: NH₄OH (concentrated), sp. gr. 0.90, 56.6%.
- 7.6 <u>Ammonium sulfate</u>, 200mg/mL: Dissolve 20 g of (NH₄)₂SO₄ in water and dilute to 100mL.
- 7.7 Ammonium sulfide 2%: Dilute 10mL (NH₄)₂S, (20-24%), to 90 mL water; total volume 100mL.

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- 7.8 <u>Barium carrier</u>, 0.00339 mg/mL, dilute 0.5 mL 20,000 ug/mLstandard to 50 mL with DI waer.
- 7.9 <u>Citric acid</u>, 1M: Dissolve 192 g of C₆H₈O₇·H₂O in water and dilute to 100mL.
- 7.10 <u>DTPA (0.2M)</u>—Diethylenetriaminepentaacetic Acid, Pentasodium salt (40% in 10). Dilute 200 mL of 40% DTPA solution to 2000 mL using DI water
- 7.11 <u>Hydrochloric acid</u>, 12M concentrated, 37.2%. Caution: Hydrochloric acid is a corrosive. Liquid and mist cause severe burns to all body tissue.
- 7.12 Hydrofluoric acid, concentrated.
- 7.13 <u>Lead carrier</u>, 15mg/mL: Dissolve 2.397g Pb(NQ)₂ in water, add 0.5 mL 16<u>N</u> HNO₃ and dilute to 100 mL with water.
- 7.14 <u>Lead carrier</u>, 1.5mg/mL: Dilute 10 mL lead carrier, (15mg/nL), to 100 mL with water.
- 7.15 Methyl orange indicator 0.1%: Dissolve 01 g methyl orange indicator in 100mL water.
- 7.16 <u>Nitric acid</u>, 16N: HNO₃ (concentrated), specific gravity 1.42, 704%.
- 7.17 Sodium hydroxide 10N: dissolve 40 g of NaOH in water and dilute to 100mL.
- 7.18 Sodium Sulfate 100 g of Na₂SO₄ to 1 L DI water (0.7m)
- 7.19 Sulfuric acid 18N: Cautiously mix 1 volume $3\underline{M}$ H₂SO₄ (conc.) with 1 volume of water.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Samples may be collected in glass or plastic containers.
- 8.3 Aqueous samples are preserved with nitric acid to a pH oless than 2.
 - 8.3.1 The pH of aqueous samples is checked upon receipt by the Sample Control Department. The pH does not require rechecking prior to analysis.
 - 8.3.2 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require a check of the pH prior to analysis.

9.0 QUALITY CONTROL

9.1 Batch

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised

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- of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples
- 9.1.3 For this analysis, batch QC consists of a <u>method blank</u>, a <u>Laboratory Control Sample</u> (LCS), and <u>Sample Duplicate</u>. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
 - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.
- 9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 Method Blank

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For Water analyses, the method blank is comprised of DI water.
- 9.2.4 For Soil analyses, tracer is added to a digestion tube and processed as the samples in Section 11.

9.3 **Laboratory Control Sample**

- 9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 For Water analyses, the LCS is comprised of DI water fortified with radium-226.
- 9.3.4 For Soil analyses, tracer and spike are added to a digestion tube and processed as the samples in Section 11.

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
- 9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate**

- 9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
- 9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See

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SOP ST-QA-0036 for details regarding the NCM process.

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Balance and thermometer calibration must be checked daily when used. Refer to SOP ST -QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes Procedure.
- See the analytical SOP for instrument calibration; ST -RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System."

11.0 PROCEDURE

11.1 Waters:

- 11.1.1 If sediment is present in the sample, this procedure requires that the sample not be shaken. If there is sediment in the sample filter an aliquot through a 0.7 um filter paper. Transfer an aliquot of the sample (typically 1 liter) to an appropriate size beaker. Label beaker with sample ID number and volume. Record all atta on sample worksheet.
 - 11.2.1.1 NOTE: The sample volume may vary according to the contract required detection limits. Review the Quality Assurance Summary for additional information.
- 11.1.2 Add 5 mL lead carrier (15 mg/mL) per liter of sample analyzed, 1.0 mL **0.00339 mg/mL barium carrier**, and 1 mL Ba-133 tracer which has an activity concentration in the range between 1000 dpm/mL to 2200 dpm/mL. Add spike standard to LCS (s) and MS (if client has requested analysis of an MS).

11.2 Soils:

- 11.2.1 Prepare per SOP ST-RC-0003, "Drying and Grinding of Soil and Solid Samples" and weigh 0.5 to 1 g into a labeled crucible.
- 11.2.2 Place in ovenat 600°C and allow to muffle for 4 hours. Allow to cool.
- 11.2.3 Transfer to digestion tube using 4M HNQ
- 11.2.4 Add 1 mL Ba-133 and 1 mL Ba 0.00339 to samples and QC. Addradium-226 spike to LCS and MS/MSD, if applicable.
- 11.2.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated hydrofluoric acid.
- 11.2.6 Digest in mod block at >119C until dry.
- 11.2.7 Carefully add 10mL nitricacid, 10 mL hydrochloric acid and 5mL hydrofluoric acid.
- 11.2.8 Digest in mod block until dry.
- 11.2.9 Carefully add 5mL nitric acid and 5mL hydrochloric acid.
- 11.2.10 Digest in mod block until dry.
- 11.2.11 Dissolve with 10mL HNO₃ and 10 mL HCl, return to mod block for 30 minutes.
- 11.2.12 Transfer to 400 mL beakers with 4M HNQ. Dilute to 200 mL with DI water.
- 11.2.13 Add 5 mL lead carrier (15mg/mL)

11.3 Precipitation:

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- 11.3.1 Add 1M citric acid in ratio of 5 m L per liter. Add methyl orange indicator until the persistence of a red color. Mix thoroughly.
- 11.3.2 Place the samples on a hotplate and heat to incipient boiling. Maintain the samples at this temperature for at least 10 minutes.
- 11.3.3 Remove the beakers form the hotplate. Caution: wear a face shield while performing the following steps as pattering may occur with the addition of sulfuric acid.
 - 11.3.3.1 If the methyl orange indicator has decomposed during the heating process add additional methyl orange to each sample as you begin this next step.
- 11.3.4 While stirring **carefully** add 15 M ammonium hydroxide until a de finite yellow color is obtained.
- 11.3.5 Add 5 mL ammonium sulfate (200 mg/nL) for each liter of sample.
- 11.3.6 Precipitate lead and barium sulfates **carefully** by stirring and slowly adding 18M sulfuric acid drop-wise until the red color reappears add approximately 2mL extra.
- 11.3.7 Place the samples back on the hotplate and heat for an additional 15 minutes. The precipitate will begin **o** settle and the solution will clear.
- 11.3.8 Remove the samples form the hotplate Cool the samples for at least 4 hours allow the precipitate to settle to the bottom of the beaker Decant the supernatantto an acid waste receptacle taking care to avoid distubing the precipitate.
- 11.3.9 Quantitatively transferthe precipitate to a 50 mL centrifuge tube, taking care to rinsche last particles out of the beaker with DI water. Centrifuge and carefully decant the supernatant to an acid waste receptacle Repeat this sep until all the precipitate is collected.
- 11.3.10 Wash the precipitate with 10 mL 16N HNO₃, vortex well to break up the ppt, centrifuge for 5 minutes at 2000 rpm, and discard the supernate to acid waste
 - 11.3.10.1 If the samples have a larger precipitte than the QC samples repeat previous step. This step may be repeated a third time if the sample contains a large amount of precipitate.
- 11.3.11 Wash the precipitate with 10mL DI water, vortex, centrifuge, and discardsupernate to acid waste.
 - 11.3.11.1 If any sample contains alarger precipitate volume than the QC samples, repeat this water wash using 20 mL of DI water. If the sample still contains more precipitate than the QC samplesconsult the supervisorbefore proceeding.
- 11.3.12 Add 10 mL of 0.2M DTPA, vortex thoroughly to break up the precipitate pellet at the bottom of the tube. Place the tubes in hot water bath (approximately 80°C) for 5-10 minutes. Remove the samples from the hebath, allow the samples to cool. When cool tighten the cap on the tube and vortexagain. Examine the centrifuge tube to see if all the precipitate is dissolved.
 - 11.3.12.1 At this point, if insoluble solids remain in the tube check the pH of the DTPA solution. If the sample pH is greater than 10 proceed to the next step. If the pH is less than 10 add 3 drops of 10 M NaOH, s wirl the tube and recheck the pH. At this point if the pH is greater than 10, repeat this stepbefore going to the next step.
- 11.3.13 Add 0.5 mL of 2% ammonium sulfidesolution to each sampleand swirl.
 - 11.3.13.1 If a b lack precipitate does not form add 10 M NaOH dropwise until a black precipitate forms, then add 3 drops more
- 11.3.14 Centrifuge the samples at 2000 RPM for 5 minutes.
- 11.3.15 Remove the tubes from the centrifuge and add 0.5 mL of 2% ammonium ulfide solution to each tube.
 - 11.3.15.1 If a precipitate forms, continue to 11.3.16.
 - 11.3.15.2 If a precipitate does not form, add 3 drops of 10M NaOH and swirl. Continue on to 11.3.16 if precipitate formed.

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11.3.15.2.1 If a precipitate does not form, add 1 mL to 1.5 mL Pb carrier and swirl. Continue on to 11.316.

- 11.3.16 Centrifuge the sample at 2000 rpm for 5 minutes.
- 11.3.17 Any tubes to which you added the 1.5 mg/mL Pb carrier set aside and repeat previous two steps until all samples have reached this point.
- 11.3.18 Place a 0.45um Pall Acrodisc, HT Tuffryn, syringe filter on a 20 mL syringe. Label new centrifuge tubes. Transfer the solution from the centrifuge tube into the syringe and filter into the new centrifuge tubes.
- 11.3.19 Cap the tubes with the sample and place in an ice bath until chilled approx. 15 minutes or longer.
- 11.3.20 Remove the samples form the ice bath , in a fume hood add 0.5 mL of 0. 00339 mg/mL barium carrier to each sample and swirl. Add 3 mL of sodium sulfateto each sample, swirl, then add 5 mL of 50% acetic acid to each sample. Replace the cap on the centrifuge tube and mix well.
- 11.3.21 Place the centrifuge tubes in the ice bath for at least 30 minutes.
- 11.3.22 While the samples are chilling write the sample ID on the back of the F-2 plastic flat disks and the petri dishes. On the petri dish also write the test "Re226".
- 11.3.23 Place the E ichrom Resolve filters on the suction filter base and assemble the filtration apparatus using the polysulfone filter funnels. Wet each filter with approximately 5 mL 80% ethanol. Let the system stand for a minute to test the filter apparatus for leaks the n apply vacuum to drain the funnels. Using DI water rinse the filters with 2 mL of DI water.
- 11.3.24 Place the petri dishes in front of the appropriate funnel to track the identity of each sample to be added to the filter funnel.
- 11.3.25 Pour the sample into the funne l. Rinse the sides of the centrifuge tube with a few mL of water and set aside.
- 11.3.26 When a sample has passed through the funnel, swirl the appropriate centrifuge tube containing the water rinse and add to the appropriate filter funnel. Cap and discard the centrifuge tube.
- 11.3.27 When this rinse has passed through the filter rinse the funnel with a few mL of DI water.
- 11.3.28 After the water has passed through all the funnels turn off the vacuum Remove the funnels and place them in the soak tank for cleaning.
- 11.3.29 Carefully remove each filter using a forceps by holding only the edge of the filter Place the filter into its respective labeled petri dish.
- 11.3.30 Place the petri dishes with their lidopened near a heat lamp to dry the filters.
- 11.3.31 Once filters are dry, remove the tape from a numbered plastic F-2 disk and carefully place the filter onto the center of the taped disk. Using the forc eps carefully touch the outside edge of the filter making sure that the edge of the filteradhere to the disk.
- 11.3.32 Replace each disk in the petri dish and place near the heat lamp until all filters have been mounted on their respective F2 disk.
- 11.3.33 Cover the petri dishes and bring them to the count room with the sample preparation paperwork.

11.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 There are no calculations pertaining to this sample preparation procedure.
- 12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

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13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- Data assessment does not pertain to this sample preparation procedure.
- Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.
- 13.3 See Analytical SOP ST-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System."

14.0 METHOD PERFORMANCE

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-OAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANGEMENT AND POLLUTION PREVENTION

- All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

6.2.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

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16.2.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for dispad by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 Radium 228 in Drinking Water, Method 904.0<u>Prescribed Procedures for Measurement of Radioactivity in Drinking Water</u> Section 8, EPA 600/430-032 (1980).
- 17.2 Percival, D. R. and Martin, D. B., "Sequential Determination of Radium-226, Radium-228, Actinium-227, and Thorium Isotopes in Environment and Process Waste Samples,"Analytical Chemistry, 461742-2749, (1974).
- 17.3 Sill, C.W, Determination of Ra-226 in Ores, Nuclear Wastes, and Environmental Samples By High Resolution Alpha Spectrometry. <u>Nuclear Chemical Waste Managament</u>, 7(3-4), pp.239-256 (1987).
- 17.4 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.6 TestAmerica Policy CA-Q-S-001, Acceptable Manual Integration Practices
- 17.7 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
- 17.8 Associated SOPs:
 - 17.7.1 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis
 - 17.7.2 ST-RC-0003, "Drying and Grinding of Soil and Solid Samples"
 - 17.7.3 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
 - 17.7.4 ST-RD-0403, Daily Calibration Verification and Maintenancof the Low Background Gas Flow Proportional Counting System
 - 17.7.5 ST-PM-0002, Sample receipt and Chain of Custody
 - 17.7.6 ST-QA-0002, Standard and Reagent Preparation
 - 17.7.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process
 - 17.7.8 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes

18.0 CHANGES FROM THE PREVIOUS REVISION

- 18.1 No Changes, Annual Review.
- 18.2 Rev. 3;
 - 18.2.1 Updated sample collection, preservation and storage times in section 8.0.
 - 18.2.2 Removed 11.1.1 regarding acidifying samples if pH is less than 2.
- 18.3 Rev. 4:
 - 18.3.1 Updated mod block temperature for soil digestion in section 11.2.

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	18.3.2	Updated precipitation procedure throughout section 11.3.
18.4	Rev.5	
	18.4.1	Grammatical errors fixed throughout SOP
	18.4.2	Removal of references to QuantIMS and Clouseau
	18.4.3	Section 11.3.28 Deleted release the vacuum by reopening the filtering apparatus stopcocks
	18.4.4	Updated section 15
18.5	Rev. 6:	
	18.5.1	Grammatical errors fixed through out SOP